

PCT

FEE CALCULATION SHEET Annex to the Request

For receiving Office use only

International application No.

Date stamp of the receiving Office

Applicant's or agent's
file reference

28030-00004

Applicant

B.C. CHEMICALS LTD. et al.

CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE

\$200.00

T

2. SEARCH FEE

\$2088.00

S

International search to be carried out by

(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search.)

3. INTERNATIONAL FEE

Basic Fee

The international application contains 23 sheets.

first 30 sheets

\$721.00

b1

0

x \$17.00

=

0.00

b2

remaining sheets

additional amount

Add amounts entered at b1 and b2 and enter total at B

\$721.00

B

Designation Fees

The international application contains 77 Max designations.

10

x

\$166.00

=

\$1660.00

D

number of designation fees

amount of designation fee

payable (maximum 10)

Add amounts entered at B and D and enter total at I

\$2381.00

I

(Applicants from certain States are entitled to a reduction of 75% of the international fee. Where the applicant is (or all applicants are) so entitled, the total to be entered at I is 25% of the sum of the amounts entered at B and D.)

4. FEE FOR PRIORITY DOCUMENT (if applicable)

\$45.00

P

5. TOTAL FEES PAYABLE

\$4714.00

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

TOTAL

☐ The designation fees are not paid at this time.

MODE OF PAYMENT

☐ authorization to charge
deposit account (see below)

☐ bank draft

☐ coupons

☒ cheque

☐ cash

☐ other (specify):

☐ postal money order

☐ revenue stamps

DEPOSIT ACCOUNT AUTHORIZATION (this mode of payment may not be available at all receiving Offices)

The RO/ ☐ is hereby authorized to charge the total fees indicated above to my deposit account.

☐ (this check-box may be marked only if the conditions for deposit accounts of the receiving Office so permit) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.

☐ is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account.

Deposit Account No.

Date (day/month/year)

Signature

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference
(if desired) (12 characters maximum)

28030-00004

Box No. I TITLE OF INVENTION

METHOD FOR THE PREPARATION OF PHYTOSTEROLS FROM TALL OIL PITCH

Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

B.C. CHEMICALS LTD.
P.O. Box 6000
Prince George, British Columbia
Canada V2N 2K3

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State (that is, country) of nationality:

CA

State (that is, country) of residence:

CA

This person is applicant
for the purposes of:

☐ all designated
States

☒ all designated States except
the United States of America

☐ the United States
of America only

☐ the States indicated in
the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

WONG, Alfred
3047 West 6th Avenue
Vancouver, British Columbia
Canada V6K 1X4

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box
is marked, do not fill in below.)

State (that is, country) of nationality:

CA

State (that is, country) of residence:

CA

This person is applicant
for the purposes of:

☐ all designated
States

☐ all designated States except
the United States of America

☒ the United States
of America only

☐ the States indicated in
the Supplemental Box

☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf
of the applicant(s) before the competent International Authorities as:

☒ agent

☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

TURLOCK, Lance A.
c/o Davis & Company
2800 Park Place - 666 Burrard Street
Vancouver, British Columbia
Canada V6C 2Z7

Telephone No.

(604) 687-9444

Facsimile No.

(604) 687-1612

Teleprinter No.

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Continuation of Box N . III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

NORMAN, Hugh S. O.
864 Reid Crescent
Prince George, British Columbia
Canada V2N 3W8

This person is:

- ☐ applicant only
☒ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

CA

State (that is, country) of residence:

CA

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☒ the United States of America only

☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

MACMILLAN, Angus Kirke
13 Sherwood Place
Tsawwassen, British Columbia
Canada V4L 2C7

This person is:

- ☐ applicant only
☒ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

CA

State (that is, country) of residence:

CA

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☒ the United States of America only

☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☐ the United States of America only

☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only
☐ applicant and inventor
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

☐ all designated States

☐ all designated States except the United States of America

☐ the United States of America only

☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.

Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

Regional Patent

- ☒ **AP** ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ **EA** Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ **EP** European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ **OA** OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line)

National Patent (if other kind of protection or treatment desired, specify on dotted line):

- | | |
|---|---|
| <input checked="" type="checkbox"/> AL Albania | <input checked="" type="checkbox"/> LS Lesotho |
| <input checked="" type="checkbox"/> AM Armenia | <input checked="" type="checkbox"/> LT Lithuania |
| <input checked="" type="checkbox"/> AT Austria | <input checked="" type="checkbox"/> LU Luxembourg |
| <input checked="" type="checkbox"/> AU Australia | <input checked="" type="checkbox"/> LV Latvia |
| <input checked="" type="checkbox"/> AZ Azerbaijan | <input checked="" type="checkbox"/> MD Republic of Moldova |
| <input checked="" type="checkbox"/> BA Bosnia and Herzegovina | <input checked="" type="checkbox"/> MG Madagascar |
| <input checked="" type="checkbox"/> BB Barbados | <input checked="" type="checkbox"/> MK The former Yugoslav Republic of Macedonia |
| <input checked="" type="checkbox"/> BG Bulgaria | <input checked="" type="checkbox"/> MN Mongolia |
| <input checked="" type="checkbox"/> BR Brazil | <input checked="" type="checkbox"/> MW Malawi |
| <input checked="" type="checkbox"/> BY Belarus | <input checked="" type="checkbox"/> MX Mexico |
| <input type="checkbox"/> CA Canada | <input checked="" type="checkbox"/> NO Norway |
| <input checked="" type="checkbox"/> CH and LI Switzerland and Liechtenstein | <input checked="" type="checkbox"/> NZ New Zealand |
| <input checked="" type="checkbox"/> CN China | <input checked="" type="checkbox"/> PL Poland |
| <input checked="" type="checkbox"/> CU Cuba | <input checked="" type="checkbox"/> PT Portugal |
| <input checked="" type="checkbox"/> CZ Czech Republic | <input checked="" type="checkbox"/> RO Romania |
| <input checked="" type="checkbox"/> DE Germany | <input checked="" type="checkbox"/> RU Russian Federation |
| <input checked="" type="checkbox"/> DK Denmark | <input checked="" type="checkbox"/> SD Sudan |
| <input checked="" type="checkbox"/> EE Estonia | <input checked="" type="checkbox"/> SE Sweden |
| <input checked="" type="checkbox"/> ES Spain | <input checked="" type="checkbox"/> SG Singapore |
| <input checked="" type="checkbox"/> FI Finland | <input checked="" type="checkbox"/> SI Slovenia |
| <input checked="" type="checkbox"/> GB United Kingdom | <input checked="" type="checkbox"/> SK Slovakia |
| <input checked="" type="checkbox"/> GD Grenada | <input checked="" type="checkbox"/> SL Sierra Leone |
| <input checked="" type="checkbox"/> GE Georgia | <input checked="" type="checkbox"/> TJ Tajikistan |
| <input checked="" type="checkbox"/> GH Ghana | <input checked="" type="checkbox"/> TM Turkmenistan |
| <input checked="" type="checkbox"/> GM Gambia | <input checked="" type="checkbox"/> TR Turkey |
| <input checked="" type="checkbox"/> HR Croatia | <input checked="" type="checkbox"/> TT Trinidad and Tobago |
| <input checked="" type="checkbox"/> HU Hungary | <input checked="" type="checkbox"/> UA Ukraine |
| <input checked="" type="checkbox"/> ID Indonesia | <input checked="" type="checkbox"/> UG Uganda |
| <input checked="" type="checkbox"/> IL Israel | <input checked="" type="checkbox"/> US United States of America |
| <input checked="" type="checkbox"/> IN India | <input checked="" type="checkbox"/> UZ Uzbekistan |
| <input checked="" type="checkbox"/> IS Iceland | <input checked="" type="checkbox"/> VN Viet Nam |
| <input checked="" type="checkbox"/> JP Japan | <input checked="" type="checkbox"/> YU Yugoslavia |
| <input checked="" type="checkbox"/> KE Kenya | <input checked="" type="checkbox"/> ZW Zimbabwe |
| <input checked="" type="checkbox"/> KG Kyrgyzstan | |
| <input checked="" type="checkbox"/> KP Democratic People's Republic of Korea | |
| <input checked="" type="checkbox"/> KR Republic of Korea | |
| <input checked="" type="checkbox"/> KZ Kazakhstan | |
| <input checked="" type="checkbox"/> LC Saint Lucia | |
| <input checked="" type="checkbox"/> LK Sri Lanka | |
| <input checked="" type="checkbox"/> LR Liberia | |

Check-boxes reserved for designating States (for the purposes of a national patent) which have become party to the PCT after issuance of this sheet:

- ☐
- ☐
- ☐

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)

Box No. VI PRIORITY CLAIM		<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.		
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application: regional Office	international application: receiving Office
item (1) 20 February 1998 (20.02.98)	2,230,373	CA		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s): (1)

* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA)
(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):

ISA /

Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority):

Date (day/month/year)

Number

Country (or regional Office)

Box No. VIII CHECK LIST; LANGUAGE OF FILING

This international application contains the following number of sheets:

request : 4
description (excluding sequence listing part) : 13
claims : 4
abstract : 1
drawings : 1
sequence listing part of description : _____

Total number of sheets : 23

This international application is accompanied by the item(s) marked below:

1. ☒ fee calculation sheet
2. ☐ separate signed power of attorney
3. ☐ copy of general power of attorney; reference number, if any:
4. ☐ statement explaining lack of signature
5. ☐ priority document(s) identified in Box No. VI as item(s):
6. ☐ translation of international application into (language):
7. ☐ separate indications concerning deposited microorganism or other biological material
8. ☐ nucleotide and/or amino acid sequence listing in computer readable form
9. ☐ other (specify):

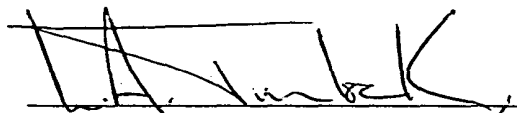
Figure of the drawings which should accompany the abstract:

Language of filing of the international application:

English

Box No. IX SIGNATURE OF APPLICANT OR AGENT

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).



Lance A. Turlock
Patent Agent

For receiving Office use only		2. Drawings: <input type="checkbox"/> received: <input type="checkbox"/> not received:
1. Date of actual receipt of the purported international application:		
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:		
4. Date of timely receipt of the required corrections under PCT Article 11(2):		
5. International Searching Authority (if two or more are competent): ISA /	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.	

Date of receipt of the record copy by the International Bureau:

For International Bureau use only

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

TURLOCK, Lance A.
Davis & Company
2800 Park Place
666 Burrard Street
Vancouver, B.C. V6C 2Z7
CANADA

PCT

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL PRELIMINARY
EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing
(day/month/year) 02.06.2000

Applicant's or agent's file reference
28030-00004

IMPORTANT NOTIFICATION

International application No.
PCT/CA99/00150

International filing date (day/month/year)
19/02/1999

Priority date (day/month/year)
20/02/1998

Applicant
B.C. CHEMICALS LTD. et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/



European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Brell, S

Tel. +49 89 2399-7271



**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/CA99/00150

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1,2,5-13	as originally filed			
3,3a,4	as received on	27/03/2000	with letter of	24/03/2000

Claims, No.:

6 (part),7-13	as received on	27/09/1999	with letter of	24/09/1999
1-5,6 (part), 14-26	as received on	27/03/2000	with letter of	24/03/2000

Drawings, sheets:

1/1	as originally filed
-----	---------------------

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference 28030-00004	<div style="display: flex; justify-content: space-between;"> <div> FOR FURTHER ACTION </div> <div> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416) </div> </div>	
International application No. PCT/CA99/00150	International filing date (day/month/year) 19/02/1999	Priority date (day/month/year) 20/02/1998
International Patent Classification (IPC) or national classification and IPC C07J9/00		
Applicant B.C. CHEMICALS LTD. et al.		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 4 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 7 sheets.</p>		
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"> I <input checked="" type="checkbox"/> Basis of the report II <input type="checkbox"/> Priority III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability IV <input type="checkbox"/> Lack of unity of invention V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement VI <input type="checkbox"/> Certain documents cited VII <input type="checkbox"/> Certain defects in the international application VIII <input type="checkbox"/> Certain observations on the international application 		
Date of submission of the demand 15/09/1999	Date of completion of this report 02.06.2000	
Name and mailing address of the international preliminary examining authority: <div style="display: flex; align-items: center;"> <div> European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 </div> </div>	Authorized officer Rudolf, M Telephone No. +49 89 2399 8604	



**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/CA99/00150

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims 1-25
	No: Claims 26
Inventive step (IS)	Yes: Claims 1-25
	No: Claims 26
Industrial applicability (IA)	Yes: Claims 1-26
	No: Claims

2. Citations and explanations

see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/CA99/00150

To section V:

Novelty:

MALIK, LUBOMIR ET AL: 'Isolation of phytosterols from tall - oil rosin' describes a process for the isolation of phytosterols, said process involving four distillation steps which are carried out in recombinant parallel paths (e.g. the light fraction of the second distillation is combined with the bottom fraction of a third distillation, and that combined fractions are re-distilled).

The present process is distinguished from said process in that it involves a sequence of distillation steps which are carried out in series, and in that the fractions are not mixed prior to further treatment.

Thus the processes as defined in claims 1-25 are novel in view of the prior art process.

Claim 26: Phytosterols are well known in the art. Claim 26 does not contain any technical feature that would allow to distinguish the claimed compositions from the phytosterols described in the prior art. The fact that the phytosterols are obtained by a novel process does not bring about novelty (or inventive step) of the phytosterols per se.

Inventive step:

Closest prior art appears to be the process described in Malik et al. referred to above. Said prior art process results in a recovery of phytosterols of 80-90% by weight. The present process is advantageous over the aforementioned prior art process since it avoids parallel distillations and uses fewer distillation steps, whereas the percentage of recovered phytosterols is equally high (about 90%, cf. table 2). Such improvement is not deducible from the prior art, an inventive step can therefore be acknowledged.

PATENT PGT/CA99/00150

In U.S. Patent No. 5,097,012 granted on 17 March 1992, Thies et al. disclose a method for the isolation of sterols from crude tall oil by water extraction at elevated temperatures and pressures.

5 In U.S. Patent No. 3,943,117 granted on 9 March 1976, Force discloses a process for saponifying tall oil pitch in which a water-soluble cationic amine is used in conjunction with an alkali. In U.S. Patent No. 4,524,024 granted on 18 June 1985, Hughes teaches the hydrolysis of tall oil pitch at elevated temperatures to increase the recovery of fatty acids from tall oil pitch. In U.S. Patent No. 3,887,537 granted on 3 June 1975, Harada et al. disclose the recovery of fatty acids and rosin acids from tall oil pitch by first
10 saponifying tall oil pitch with an alkali metal base and a low molecular weight alcohol, and then introducing the reacted mixture into a thin film evaporator to remove low-boiling matter such as water, alcohol use and light unsaponifiables. The bottom fraction from the first evaporator is next fed to a second thin film evaporator in which the unsaponifiables including sterols are removed as the light ends and a molten soap is recovered as the
15 bottom fraction. Fatty acids and rosin acids are recovered from the molten soap fraction by acidulation conventionally with a mineral acid. In U.S. Patent No. 3,926,936 granted on 16 December 1975, Lehtinen teaches the recovery of fatty acids and rosin acids from tall oil pitch by reacting tall oil pitch with an alkali at 200 to 300 degrees Celsius, in the amount of 5 to 25% of tall oil pitch, prior to vacuum distillation of the heated mixture to
20 recover the fatty acids and rosin acids in the distillate fraction.

Reference is also made to Chemical Abstracts, vol. 112, no. 20, 14 May 1990, Columbus, Ohio US; abstract no. 181758, MALIK, Lubomir et al: "Isolation of phytosterols from tall - oil rosin", XP002104877 & CS 256 092 A (Czech). Malik et al. disclose a process for extracting phytosterols which includes the use of four distillation
25 stages. Product flow is split into parallel distillation paths in a first distillation stage then, following further distillation in each parallel path, is partially recombined prior to a final distillation stage. To achieve high purity phytosterols, the output from the final distillation

PCT/CA99/00150

stage is subjected to two stages of crystallization utilizing relatively large amounts of solvent.

SUMMARY OF THE INVENTION

In a broad aspect of the present invention there is provided a new and improved method of preparing phytosterols from tall oil pitch containing steryl esters, the method comprising the steps of:

- (a) converting the steryl esters to free phytosterols while in the pitch to produce a modified pitch containing the free phytosterols;
- (b) distilling the modified pitch in a first evaporator to remove light ends from the modified pitch and produce a bottom fraction containing the free phytosterols;
- (c) distilling only the bottom fraction in a second evaporator to produce a light phase distillate containing the free phytosterols;
- (d) dissolving only the light phase distillate in a solvent comprising an alcohol to produce a solution containing the free phytosterols;
- (e) cooling the solution to produce a slurry with the free phytosterols crystallized in the slurry; and,
- (f) washing and filtering the slurry to isolate the crystallized phytosterols.

Preferably, the step of converting the steryl esters to free phytosterols comprises the steps of saponifying the tall oil pitch with an alkali metal base, neutralizing the saponified pitch with an acid, and heating the neutralized pitch to remove water. The resulting pitch with such water removed defines the modified pitch.

Unlike the process of Malik et al., the foregoing process enables the preparation of high purity phytosterol crystals from tall oil pitch with only two distillation stages and only one stage of crystallization, and to do so with the use of a comparatively small amount of solvent. Nevertheless, it may be considered desirable in some cases to achieve phytosterol yields with even higher crystal purity. In accordance with another embodiment of the invention, a marginal improvement is achieved as follows:

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- (a) producing a light phase distillate containing free phytosterols in the manner described in steps (a) to (c) above;
- (b) re-distilling only the light phase distillate so produced to enhance the concentration of free phytosterols in the light phase distillate;
- 5 (c) dissolving only the re-distilled light phase distillate in a solvent comprising an alcohol to produce a solution containing the free phytosterols; and,
- (d) continuing the procedure as in steps (d) and (f) above to isolate crystallized phytosterols.

Although this procedure involves additional distillation steps, the amount of
10 alcohol required during the crystallization stage remains small compared to the case of Malik et al.

BRIEF DESCRIPTION OF THE DRAWINGS

The FIGURE shows a schematic flow diagram for the preparation of high purity phytosterol crystals from tall oil pitch in accordance with the present invention.

15 DESCRIPTION OF PREFERRED EMBODIMENT

In accordance with the present invention, the isolation of phytosterols from tall oil pitch first requires converting steryl esters present in the pitch to free phytosterols while in the pitch. The result is a modified pitch containing free phytosterols.

It is contemplated that the required conversion may be accomplished by various
20 methods. In the FIGURE, the conversion step is indicated by block 30 (shown in broken outline) which receives an incoming feed of tall oil pitch 1 and produces modified pitch 11 as an output. The presently preferred method of conversion involves the use of an alkali base treatment and is indicated by the elements contained within block 30.

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WE CLAIM:

1. A method of preparing phytosterols from tall oil pitch (1) containing steryl esters, said method comprising the steps of:

- 5 (a) converting said steryl esters to free phytosterols while in said pitch to produce a modified pitch (11) containing said free phytosterols;
- (b) distilling said modified pitch (11) in a first evaporator (12) to remove light ends (13) from said modified pitch and produce a bottom fraction (14) containing said free phytosterols;
- 10 (c) distilling only said bottom fraction (14) in a second evaporator (15) to produce a light phase distillate (16) containing said free phytosterols;
- (d) dissolving only said light phase distillate (16) in a solvent (21) comprising an alcohol to produce a solution containing said free phytosterols;
- (e) cooling said solution to produce a slurry (19) with said free phytosterols crystallized in said slurry; and,
- 15 (f) washing and filtering said slurry (19) to isolate said crystallized phytosterols (22).

2. A method as defined in claim 1, wherein said modified pitch (11) comprises less than 1% water by weight.

20 3. A method as defined in claim 1 or 2, wherein said solvent (21) comprises a low molecular weight monohydric alcohol.

4. A method as defined in claim 1 or 2, wherein said solvent (21) comprises a low molecular weight monohydric alcohol and water.

5. A method as defined in claim 1 or 2, wherein said slurry (19) is washed and filtered using a solvent like said solvent (21) used to dissolve said light phase distillate.

25 6. A method as defined in claim 1, wherein said step of converting said steryl esters to free phytosterols comprises the steps of:

- (a) saponifying said tall oil pitch (1) with an alkali metal base (2);

- (b) neutralizing said saponified pitch with an acid (5); and,
- (c) heating said neutralized pitch to remove water, the resulting pitch with such water removed defining said modified pitch (11).

7. A method as defined in claim 6, wherein said alkali metal base (2) is selected from the group consisting of:

- (a) sodium hydroxide;
- (b) potassium hydroxide;
- (c) sodium hydroxide and potassium hydroxide:

8. A method as defined in claim 7, wherein in the weight percentage of alkali metal base (2) to tall oil pitch (1) is in the range of 1% to 15%.

9. A method as defined in claim 7, wherein said saponification is conducted at a temperature in the range of 100 to 250 deg. C for a period in the range of 60 to 300 minutes.

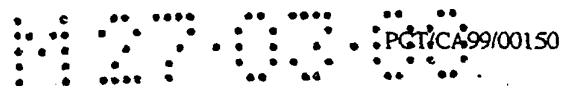
10. A method as defined in claim 6, wherein said acid (5) is an organic acid.

11. A method as defined in claim 6, wherein said acid (5) is a mineral acid.

12. A method as defined in claim 11, wherein said mineral acid (5) is selected from the group consisting of:

- (a) sulphuric acid;
- (b) hydrochloric acid;
- (c) phosphoric acid;
- (d) a combination of acids comprising two or more of sulphuric acid, hydrochloric acid and phosphoric acid.

13. A method as defined in claim 6, wherein said neutralization is conducted at a temperature in the range of 10 to 100 deg. C for a period in the range of 1 to 10 hours.



14. A method as defined in claim 6, wherein said neutralized pitch has a water phase pH in the range of 4 to 7.
15. A method as defined in claim 6, wherein said heating step comprises heating at a temperature in the range 90 to 100 deg. C for a time sufficient to effect the bulk
5 disengagement of water from the organic phase.
16. A method as defined in claim 15, wherein said heating step further comprises heating under vacuum conditions such that said modified pitch (11) comprises less than 1% water by weight.
17. A method as defined in claim 1 or 6, wherein said light ends are removed in a
10 wiped film evaporator (12) operating at a pressure in the range of 0.1 to 10 millibars and at a temperature in the range 160 to 280 deg. C.
18. A method as defined in claim 1 or 6, wherein said bottom fraction is evaporated in a wiped film evaporator (15) operating at a pressure in the range of 0.01 to 1.0 millibars and at a temperature in the range 180 to 300 deg. C.
- 15 19. A method as defined in claim 6, wherein said solvent (21) comprises a low molecular weight monohydric alcohol.
20. A method as defined in claim 6, wherein said solvent (21) comprises a low molecular weight monohydric alcohol and water.
21. A method as defined in claim 1 or 6 in which the crystallization of phytosterols is
20 effected at a temperature in the range of 0 to 35 deg. C.
22. A method of preparing phytosterols from tall oil pitch (1) containing steryl esters, said method comprising the steps of:
- (a) converting said steryl esters to free phytosterols while in said pitch to produce a modified pitch (11) containing said free phytosterols;

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- (b) distilling said modified pitch (11) in a first evaporator (12) to remove light ends (13) from said modified pitch and produce a bottom fraction (14) containing said free phytosterols;
- (c) distilling only said bottom fraction (14) in a second evaporator (15) to produce a light phase distillate (16) containing said free phytosterols;
- (d) re-distilling only said light phase distillate (16) to enhance the concentration of free phytosterols in said light phase distillate;
- (e) dissolving only said re-distilled light phase distillate in a solvent (21) comprising an alcohol to produce a solution containing said free phytosterols;
- (f) cooling said solution to produce a slurry (19) with said free phytosterols crystallized in said slurry; and,
- (g) washing and filtering said slurry (19) to isolate said crystallized phytosterols (22).
23. A method as defined in claim 22, wherein said solvent (21) further comprises water added in a proportion up to 35% by weight relative to the organic solvent phase.
24. A method as defined in claim 23, wherein the weight ratio of solvent to distillate is between 0.3 to 2.0.
25. A process according to claim 19, 20 or 24 in which the alcohol is selected from:
- (a) methanol;
- (b) ethanol;
- (c) 2-propanol;
- (d) a combination of alcohols comprising two or more of methanol, ethanol and 2-propanol.
26. Phytosterols prepared from tall oil pitch in accordance with the method as defined in any one or more of the preceding claims.

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 28030-00004	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/CA 99/ 00150	International filing date (day/month/year) 19/02/1999	(Earliest) Priority Date (day/month/year) 20/02/1998
Applicant B.C. CHEMICALS LTD. et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.



It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.



the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international search was carried out on the basis of the sequence listing:



contained in the international application in written form.



filed together with the international application in computer readable form.



furnished subsequently to this Authority in written form.



furnished subsequently to this Authority in computer readable form.



the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.



the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ Certain claims were found unsearchable (See Box I).

3. ☐ Unity of invention is lacking (see Box II).

4. With regard to the title,



the text is approved as submitted by the applicant.



the text has been established by this Authority to read as follows:

5. With regard to the abstract,



the text is approved as submitted by the applicant.



the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is Figure No.



as suggested by the applicant.



because the applicant failed to suggest a figure.



because this figure better characterizes the invention.

1



None of the figures.

A. CLASSIFICATION OF SUBJECT MATTER IPC 6 C07J9/00 C11B13/00		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 6 C07J C11B		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CHEMICAL ABSTRACTS, vol. 112, no. 20, 14 May 1990 Columbus, Ohio, US; abstract no. 181758, MALIK, LUBOMIR ET AL: "Isolation of phytosterols from tall - oil rosin" page 139; column 1; XP002104877 see abstract & CS 256 092 A (CZECH.)	1-25
Y	US 4 076 700 A (HARADA TETSUYA ET AL) 28 February 1978 see examples 1,4	1-25
Y	US 3 887 537 A (HARADA TETSUYA ET AL) 3 June 1975 see example 2	1-25
-/--		
<input checked="" type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex.		
* Special categories of cited documents : <div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>*A* document defining the general state of the art which is not considered to be of particular relevance</p> <p>*E* earlier document but published on or after the international filing date</p> <p>*L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>*O* document referring to an oral disclosure, use, exhibition or other means</p> <p>*P* document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>*&* document member of the same patent family</p> </div> </div>		
Date of the actual completion of the international search <div style="text-align: center; font-weight: bold;">4 June 1999</div>		Date of mailing of the international search report <div style="text-align: center; font-weight: bold;">25/06/1999</div>
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016		Authorized officer <div style="text-align: center; font-weight: bold;">Watchorn, P</div>

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	GB 895 145 A (EASTMAN KODAK) 2 May 1962 see page 1, line 57 - line 63 see page 2, line 9; example 1 -----	1-25
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Y	US 4 524 024 A (HUGHES RICHARD E) 18 June 1985 see column 10, line 8 - column 11, line 25 -----	1-25

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